IS 326-8 (2005): Methods of sampling and test for natural and synthetic perfumery materials (Part 8) Determination of ester value, content of esters and alcohols [PCD 18: Natural and Synthetic Fragrance Materials]

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IS 326 ( Part 8 ) : 2005
ISO 709 : 2001

*भारतीय मानक*

# प्राकृतिक और संश्लेषित सुगन्ध सामग्री के नमूने लेने और परीक्षण की पद्धतियाँ

**भाग 8 ऐस्टर मान ज्ञात करना**

*( तीसरा पुनरीक्षण )*

*Indian Standard*

# METHODS OF SAMPLING AND TEST FOR NATURAL AND SYNTHETIC PERFUMERY MATERIALS

**PART 8 DETERMINATION OF ESTER VALUE**

*( Third Revision )*

ICS 71.100.60



**BUREAU OF INDIAN STANDARDS**
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

*May* 2005

**Price Group 3**

NATIONAL FOREWORD

This Indian Standard ( Part 8 ) ( Third Revision ) which is identical with ISO 709 : 2001 'Essential oils — Determination of ester value' issued by the International Organization for Standardization ( ISO ) was adopted by the Bureau of Indian Standards on the recommendations of the Natural and Synthetic Fragrance Materials Sectional Committee and approval of the Petroleum, Coal and Related Products Division Council.

The text of ISO Standard has been proposed to be approved as suitable for publication as an Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.

b) Comma ( , ) has been used as a decimal marker while in Indian Standards, the current practice is to use a point ( . ) as the decimal marker.

In this adopted standard, reference appears to certain International Standards for which Indian Standards also exist. The corresponding Indian Standards, which are to be substituted in their places are listed below along with their degree of equivalence for the editions indicated. However, that International Standard cross-referred in this adopted ISO Standard, which has subsequently been revised, position in respect of that latest ISO Standard has been given:

| *International Standard* | *Corresponding Indian Standard* | *Degree of Equivalence* |
|---|---|---|
| ISO 385-1 : 1984 Laboratory glassware — Burettes — Part 1 : General requirements | IS 1997 : 1982 Burettes ( *second revision* ) | Equivalent |
| ISO 1242 : 1999 Essential oils — Determination of acid value | IS 326 ( Part 7 ) : 1980 Methods of sampling and test for natural and synthetic perfumery materials: Part 7 Determination of acid value ( *second revision* ) | do |

The Technical Committee responsible for the preparation of this standards will review the provisions of the following International Standard and decided that this is acceptable for use in conjunction with this standard:

| *International Standard* | *Title* |
|---|---|
| ISO 356 : 1996 | Essential oils — Preparation of test samples |

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values ( *revised* )'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Indian Standard*

# METHODS OF SAMPLING AND TEST FOR NATURAL AND SYNTHETIC PERFUMERY MATERIALS

## PART 8 DETERMINATION OF ESTER VALUE

*( Third Revision )*

## 1 Scope

This International Standard specifies a method for the determination of the ester value of an essential oil.

This method is not applicable to essential oils containing lactones or an appreciable proportion of aldehydes.

## 2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 356, *Essential oils — Preparation of test samples*

ISO 385-1, *Laboratory glassware — Burettes — Part 1: General requirements*

ISO 1242, *Essential oils — Determination of acid value*

## 3 Term and definition

For the purposes of this International Standard, the following term and definition applies.

**3.1**
**ester value**
**EV**
number of milligrams of potassium hydroxide required to neutralize the acids liberated by the hydrolysis of esters present in 1 g of the essential oil

## 4 Principle

The esters present in the essential oil are hydrolysed by heating under specified conditions with an excess of a standard volumetric ethanolic potassium hydroxide solution. The excess alkali is determined by back titration with a standard solution of hydrochloric acid.

## 5 Reagents

During the analysis, use only reagents of recognized analytical grade and distilled or demineralized water or water of equivalent purity.

**5.1 Ethanol**, 95 % (volume fraction) at 20 °C, freshly neutralized with the potassium hydroxide solution (5.2), in the presence of the coloured indicator (5.4) used for the determination.

5.2 **Potassium hydroxide**, standard volumetric ethanolic solution, $c$(KOH) = 0,5 mol/l at 20 °C, freshly restandardized before each series of tests.

5.3 **Hydrochloric acid**, standard volumetric solution, $c$(HCl) = 0,5 mol/l at 20 °C.

It is important that the reagent be taken at the specified temperature of 20 °C, particularly the ethanolic solution of potassium hydroxide, as the volume varies greatly with temperature.

5.4 **Coloured indicator.**

Use **phenolphthalein**, 2 g/l solution in ethanol (5.1), or **phenol red**, 0,4 g/l solution in ethanol, 20 % (volume fraction), if the essential oil has components that contain phenol groups.

NOTE This particular case will be specified in the specific standard for the essential oil concerned.

## 6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 **Saponification flask**, with ground glass neck, of alkali-resistant glass, of capacity 100 ml to 250 ml, to which can be fitted a ground glass air condenser at least 1 m in length with 1 cm to 1,5 cm internal diameter.

If necessary, and particularly for the essential oils with high light fractions and depending on the time placed in the boiling water bath, the glass tube may be replaced by a water-cooled reflux condenser.

6.2 **Test tubes**, of capacity 5 ml.

6.3 **Burettes**, of capacity 25 ml, graduated in 0,05 ml, complying with the requirements of ISO 385-1, class B.

6.4 **Boiling water bath**.

6.5 **Analytical balance**, accurate to the nearest 0,001 g.

6.6 **Potentiometer**.

## 7 Sampling

Sampling is not included in the method specified in this International Standard. A recommended sampling method is given in ISO 212[1)].

It is important that the laboratory receive a representative sample, not damaged or modified during transport or storage before the arrival at the laboratory.

## 8 Preparation of test sample

The test sample shall be prepared according to ISO 356.

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1) ISO 212, *Essential oils — Sampling.*

# 9 Procedure

## 9.1 Test portion

Weigh, to the nearest 0,005 g, 2 g of the test sample.

The test portion may be different from this, if so specified in the specific standard for the essential oil concerned.

## 9.2 Blank test

Carry out a blank test, in parallel with the determination (9.3), under the same conditions and using the same reagents. (See 9.3.3.)

## 9.3 Determination

**9.3.1** Introduce the test portion (9.1) into the saponification flask (6.1). Add from the burette (6.3) 25 ml of the potassium hydroxide solution (5.2) (see note) and fragments of pumice stone or porcelain.

NOTE If the test portion has been retained from the determination of the acid value, it will not be necessary to neutralize it before adding the potassium hydroxide.

For oils with a high ester value, increase the volume of the potassium hydroxide solution (5.2) used so that $(V_0 - V_1)$ (see clause 10) is at least equal to 10 ml.

For oils with a low ester value, increase the mass of the test portion used.

Attach the air condenser or water-cooled reflux condenser, and place the flask in the boiling water bath (6.4) for a time depending on the essential oil analysed. This time is mentioned in the specification for the oil to be tested.

Allow to cool and remove the tube. Add 20 ml of water and 5 drops of the phenolphthalein solution, or of the phenol red solution (5.4) if the essential oil contains phenols or compounds with phenolic groups.

**9.3.2** Titrate the excess potassium hydroxide with the hydrochloric acid (5.3).

**9.3.3** This determination may be carried out with the solution resulting from the determination of the acid value, which can be used as the blank test, by adding 5 ml of ethanol (5.1) in this blank test before the addition of the 25 ml of potassium hydroxide solution (this volume corresponds to the volume introduced during the determination of the acid value).

## 9.4 Potentiometry

Potentiometry may be used for all the essential oils, but it is particularly recommended for highly coloured essential oils for which it is difficult to appreciate the endpoint of the coloured indicator (e.g. vetiver oil). In this case, the same reagents and apparatus shall be used.

NOTE These special cases will be established in the specific standards for the essential oils concerned.

## 10 Expression of results

### 10.1 Calculation

#### 10.1.1 Ester value

The ester value (EV) is given by the formula

$$EV = \frac{28,05}{m}(V_0 - V_1) - AV$$

where

$V_0$ is the volume, in millilitres, of hydrochloric acid (5.3) used for the blank test (9.2);

$V_1$ is the volume, in millilitres, of hydrochloric acid (5.3) used for the determination (9.3.2);

$m$ is the mass, in grams, of the test portion;

AV is the acid value determined according to ISO 1242.

The mass fraction of ester, $w$, as a percentage, with respect to a stated ester, is given by the formula

$$w = \frac{M_r \cdot EV}{561}$$

where

$M_r$ is the relative molecular mass of the ester used to express the results conventionally;

EV is the ester value calculated as above.

Express the ester value to two significant figures when it is less than 100, and to three significant figures when it is 100 or more.

#### 10.1.2 Ester value determined after the acid value

When the determination is carried out on the solution resulting from the determination of the acid value, the ester value (EV) is obtained by the formula

$$EV = \frac{28,05}{m}(V_0 - V_1')$$

where $V_1'$ is the volume, in millilitres, of hydrochloric acid (5.3) used in the new determination.

## 11 Test report

The test report shall state:

— all information necessary for the complete identification of the sample;

— the sampling method used, if known;

— the test method used, with reference to this International Standard;

— all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the test result(s);

— the test result(s) obtained.

# Annex A
(informative)

# Precision

## A.1 General

At the time of publication of this International Standard, precision values for this test method have not been established. Nevertheless, taking into account the data available at present, it was considered useful for the users of this International Standard to include some indications of the values obtained for repeatability and reproducibility, and it is expected to be able to specify definitive values in the next revision of this International Standard.

## A.2 Repeatability

The absolute difference between two independent single test results, obtained using this method on the same essential oil tested in the same laboratory by the same operator using the same equipment within a short period of time, was not, in more than 5 % of cases, greater than 0,7 as ester value or 0,25 % for an ester having, for example, a molar mass of 196,29.

NOTE In the case of coloured essential oils, the difference between two measurements is greater when colorimetry is used instead of potentiometry.

## A.3 Reproducibility

The absolute difference between two single test results, obtained using the same method on the same essential oil tested in different laboratories with different operators using different equipment, was not, in more than 5 % of cases, greater than 1,4 as ester value or 0,5 % for an ester having, for example, a molar mass of 196,29.

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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards : Monthly Additions'.

This Indian Standard has been developed from Doc : No. **PCD 18 ( 2205 ).**

**Amendments Issued Since Publication**

| Amend No. | Date of Issue | Text Affected |
|---|---|---|
| | | |
| | | |
| | | |
| | | |

BUREAU OF INDIAN STANDARDS

**Headquarters:**

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110 002
*Telephones :* 2323 0131, 2323 3375, 2323 9402 *Website :* www.bis.org.in

**Regional Offices :** *Telephones*

Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110 002 — 2323 7617, 2323 3841

Eastern : 1/14 C. I. T. Scheme VII M, V. I. P. Road, Kankurgachi, KOLKATA 700 054 — 2337 8499, 2337 8561, 2337 8626, 2337 9120

Northern : SCO 335-336, Sector 34-A, CHANDIGARH 160 022 — 260 3843, 260 9285

Southern : C. I. T. Campus, IV Cross Road, CHENNAI 600 113 — 2254 1216, 2254 1442, 2254 2519, 2254 2315

Western : Manakalaya, E9 MIDC, Marol, Andheri (East), MUMBAI 400 093 — 2832 9295, 2832 7858, 2832 7891, 2832 7892

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Printed at New India Printing Press, Khurja, India